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(54) PROCESS FOR FIXING DYE-STUFFS ON CELLULOSIC MATERIALS

(71) We, FARBWERKE HOECHST AKTIENGESELLSCHAFT, vormals Meister Lucius & Brüning, a Body Corporate recognised under German Law, of 6230 Frankfurt (M)-Hoechst, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a process for the printing and continuous fixation of azo dyestuffs and, optionally, reactive or disperse dyestuffs on textile materials partially or completely comprising cellulosic material.

It is known that by printing diazotised amines in addition to, or mixed with, reactive dyestuffs, onto cotton fabrics which have beforehand been impregnated with alkaline solutions of  $\beta$ -hydroxynaphthoic acid arylides, prints can be obtained which represent an optimum as regards low price and brilliance. Additionally, a great advantage of this process is that the dyestuffs can be fixed onto the fibre by a fully continuous process. The diazotised amines are directly coupled to the naphthol derivative on the fibre to give a final dyestuff, whilst the reactive dyestuffs are fixed, for example, by a dipping process, lasting 5 seconds, in a hot alkaline solution containing very large amounts of salt. This method is especially advisable if at least 30% of the naphthol bottoming is utilised by coupling with the diazonium salts to produce a colour.

In this method, however, the various process steps (applying the naphthol derivative before the actual printing process) are a nuisance. Furthermore, only a certain limited number of colour shades is achievable with one and the same naphthol derivative which is applied to the fabric by padding and used as bottoming.

This disadvantage has already previously been overcome by developing stabilised dyeing preparations which contain, as the essential constituents, a naphthol derivative and stable diazo compounds; by printing the fabric with various of these dyeing preparations, the most diverse colour shades can thus be produced on the fibres and independence from the particular bottoming was achieved. The stable diazo compounds mentioned are, for example, diazoamino compounds which are obtained by reaction of the unstable diazonium salts with, for example, cyanamide, anthranilic acid or other amines. They are sufficiently stable in an alkaline medium that they can be combined with naphtholate solutions. It is also possible to convert the diazotised amines into anti-diazotates by means of strong alkalis, and their mixtures with naphtholates lead to similar, though somewhat less stable, dyeing preparations.

Dyeing preparations of the types mentioned have been commercially available for many years. With these preparations, the stabilised diazo compounds can be re-converted on the fibre, by means of steaming processes, into the diazotised base, whereupon coupling with the coupling component, preferably a naphthol derivative, to give the dyestuff occurs. If this steaming is carried out in an acid medium, distinctly more brilliant shades are obtained than on neutral steaming. The relevant literature recommends steaming processes of 3—5 minutes, and elsewhere 5—7 minutes, depending on the steaming equipment used. These steaming times are, however, still so long that a continuous process, for example steaming and washing in series, can hardly be carried out in practice if it is desired to achieve optimum speeds of the washing process. The long steaming times are attributable to the fact that in the case of the customary steamers of substantial volume the steam atmosphere becomes depleted in acid content in the immediate vicinity of the fabric which has been printed

of a commercial form and standard are dissolved in the cold, and dispersed, by adding 20 g of 32.5% strength sodium hydroxide solution, 50 g of ethanol, 200 g of water and 50 g of urea and the mixture is subsequently stirred into 500 g of a neutral wheat starch-tragacanth thickener and is made up with water or thickener to 1 kg of printing paste.

Using this printing ink, any desired pattern is printed by roller printing onto a 67:33 polyester/cotton mixed fabric. After drying the print, the back of the fabric is moistened, by means of a stippling roller, with an acid solution of 1 part of 50% strength acetic acid, 1 part of 85% strength formic acid and 1 part of water and is subsequently steamed for 10 seconds at 190° to 200°C in a solvent steamer. To avoid excessive condensate formation, the fabric which enters the steamer is beforehand preheated to 150°C in an infrared tunnel which precedes the steamer. Fixing takes place in the steamer with ethylene glycol vapour.

In this way, both the red azo dyestuff from the dyeing preparation is developed and fixed to the cotton constituent, and the blue dispersion dyestuff is fixed on the polyester constituent, in one process stage.

#### WHAT WE CLAIM IS:—

1. A process for the printing and continuous fixation of an azo dyestuff on a textile material partially or completely comprising cellulosic material, which comprises printing the textile material with a stabilized dyeing preparation which forms an azo dyestuff, and forming and fixing the azo dyestuff by treating the printed textile material for a period of from 5 to 15 seconds in a continuous operation with steam in the presence of one or more volatile organic acids, the acid(s) being present in such a concentration that an acidic pH is imparted to the textile material.

2. A process for the printing and continuous fixation of an azo dyestuff and a reactive dyestuff on a textile material partially or completely comprising cellulosic material, which comprises printing the textile material with a stabilized dyeing preparation which forms an azo dyestuff and with a dyeing preparation containing a reactive dyestuff, and, in a continuous operation, forming and fixing with azo dyestuff by treating the printed textile material for a period of from 5 to 15 seconds with steam in the presence of one or more volatile organic acids, the acid(s) being present in such a concentration that an acidic pH is imparted to the textile material, and subsequently fixing the reactive dyestuff by a two-phase fixation process.

3. A process as claimed in claim 2, wherein the reactive dyestuff is applied to the textile material by an overprinting process after the material has been printed with the stabilized dyeing preparation which forms an azo dyestuff.

4. A process as claimed in any one of claims 1 to 3, wherein the treatment with steam is carried out using a counter-current steaming method.

5. A process as claimed in any one of claims 1 to 4, wherein after the fixation operation(s) the textile material is subjected to a continuous finishing process comprising washing, soaping and rinsing.

6. A process as claimed in any one of claims 1 to 5, wherein the stabilized dyeing preparation which forms an azo dyestuff comprises a stabilized diazonium compound and a coupling component, which stabilized diazonium compound is reconvertable to a reactive diazotized base by the treatment with steam.

7. A process as claimed in any one of claims 1 to 6, wherein the steaming in the presence of one or more volatile organic acids is carried out by either

a) enriching the steam with acid by spraying one or more volatile organic acids into the steam, or

b) enriching the steam with acid by dripping one or more volatile organic acids onto a heated plate in the vicinity of the steam inlet of the steaming apparatus used, or

c) after the dyestuff printing, printing the textile material with a solution of one or more volatile organic acids and applying steam to the thus printed material.

8. A process as claimed in claim 7 c), wherein a stippling roller is used to apply the acid(s) to the textile material.

9. A process as claimed in any one of claims 1 to 8, wherein a volatile organic acid is acetic acid or formic acid.

10. A process as claimed in any one of claims 1 to 9, wherein the steaming operation is carried out at a temperature of from 150 to 200°C.

11. A process as claimed in any one of claims 1 to 10, wherein steaming is carried out in a steamer in which the printed textile material passes through a narrow tunnel, which may contain one or more baffles for restricting the vapour flow along the tunnel.

12. A process as claimed in any one of claims 1 to 10, wherein the steaming is carried out in a perforated-drum steamer, in which the printed textile material passes over a perforated drum and the steam is forced or drawn through the material.

5      13. A process as claimed in any one of claims 1 to 12, wherein, in the steaming operation, steam is replaced by a solvent vapour.      5

14. A process as claimed in claim 13, wherein the solvent vapour is ethylene glycol vapour.

15. A process as claimed in any one of claims 1 to 14, wherein the textile material comprises cotton or a polyester/cotton mixture.

10      16. A process as claimed in claim 1 wherein, when the textile material comprises a proportion of synthetic fibres, the textile material is also printed with a dispersion dyestuff and steaming is carried out at a temperature of at least 180°C to fix the dispersion dyestuff.      10

15      17. A process as claimed in any one of claims 1, 2 or 16, conducted substantially as described herein.      15

18. A process as claimed in claim 1, conducted substantially as described in any one of Examples 4 to 7.

19. A process as claimed in claim 2, conducted substantially as described in any one of Examples 1 to 3.

20      20. A process as claimed in claim 16, conducted substantially as described in Example 8.      20

21. A textile material partially or completely comprising cellulosic material, whenever printed by a process as claimed in any one of claims 1 to 20.

ABEL & IMRAY,  
Chartered Patent Agents,  
Northumberland House,  
303—306, High Holborn,  
London, W.C.1.

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